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ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

TECHNOLOGY CENTER

INVESTIGATION OF GLASS FIBER STRENGTH ENHANCEMENT THROUGH BUNDLE DRAWING

by

N. A. Weil and J. S. Islinger

for

Bureau of Naval Weapons
Navy Department
Washington, D. C.

prepared under
Contract No. NOw61-0259-c

ARMOUR RESEARCH FOUNDATION
of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

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FOREWORD

This is report No. 4 (Fourth Quarterly Report) on Armour Research Foundation (ARF) Project K213, covering the period of October 7, 1961 through January 6, 1962 on Contract NOw-61-0259-c. This contract is under the direct supervision of the Non-Metals Branch, Materials Division, Bureau of Naval Weapons with Mr. J. J. Gurtowski, RRMA-31, acting as the BuWeps Project Engineer.

ARF staff members who have contributed to the research discussed in the report include: Joseph S. Islinger, H. Robert Nelson, Dr. Edward L. McDowell, Rangwald S. Olsen, James Staulcup and Dr. Nicholas A. Weil.

Experimental data on this project are recorded in ARF Logbook Nos. C11280 and C11954.

Respectfully submitted,

ARMOUR RESEARCH FOUNDATION OF
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INVESTIGATION OF GLASS FIBER STRENGTH ENHANCEMENT THROUGH BUNDLE DRAWING

I. INTRODUCTION

The objective of this program is the development of a method capable of producing glass fibers with enhanced tensile strengths. The method under investigation involves the bundle drawing of inherently flaw free ultra fine fibers with a suitable coating applied for protection against abrasion and atmospheric degradation. By a reduction of the ever present flaws in glass, it is expected that marked improvements in the tensile strength of glass fibers can be obtained. To provide coated fibers for the subsequent bundle drawing operation, fibers are being drawn from glass rods in the ARF fiber drawing apparatus. Metallic coatings are applied to the virgin fibers immediately after drawing. The coated fibers are collected and bundled in a glass tube or sheath. The bundled fibers are then redrawn as a unit in order to provide fibers of extremely fine diameters.

Investigations during this fourth quarter comprised:

1. A theoretical investigation was undertaken with two aims in mind: (a) to ascertain whether theory would predict an absolute lower limit on the fiber diameter attainable in single-filament drawing operations, and (b) to determine the specific effect of various parameters upon this limiting value. The beginning of such a theoretical undertaking is presented in this report.

2. A series of fiber drawing experiments was undertaken to continue efforts to improve the metal coating procedure for glass fibers and to continue the efforts of determining the feasibility of the bundle drawing technique in producing ultra-fine glass fibers.

II. ANALYSIS OF GLASS FIBER DRAWING FROM ROD FEED

A. Introduction

In glass filament drawing operations an increase in the winding speed of the take-up drum results in the production of successively finer fibers. This is true, regardless of whether the fiber is produced by drawing from a melt through a bushing, or whether it results from a comparatively large diameter rod being fed through the hot zone of a furnace. It has also been known for a long time that, in either method of glass fiber production, there appear to be very real limitations regarding the finest fibers that can be drawn. In commercial practice, it appears that single filament fibers can be produced to a minimum diameter of about 3-4μ; with careful laboratory controls, researchers have succeeded in drawing continuous filaments as fine as 1μ. However, single filament drawing operations appear incapable of yielding continuous fibers finer than this diameter.

Yet, well known theories suggest that the strength of the fiber should rise as its diameter is reduced; at a sufficiently fine size (estimated to be represented by a diameter of about 0.05μ), the fiber should attain the theoretical strength of glass, that is, a strength assumed to range between 1-4 million psi. The validity of increasing strength with reduced size has been amply proven by numerous investigators down to sizes (1μ) capable of production with continuous filament drawing. However, the hypothesis of the attainment of near theoretical strength values could never be confirmed before, because of the limitations of existing methods for producing sufficiently fine fibers. It is, in fact, one of the principal purposes of the present program to explore whether the needed ultra-fine fibers can be produced by means of a novel concept identified as "bundle-drawing".

In the original proposal forming the basis of the current work, it was suggested that the incapability of single-filament drawing operation may be ascribed to a form of tensile instability developing during drawing, which would force rupture of the fiber below a certain critical size. Indeed, if this hypothesis were true, then it would be useless to rely upon single filament drawing operations for the production of ultra-fine fibers, and all

effort should be directed toward various bundle-drawing concepts for the attainment of this goal.

With this view, a theoretical investigation was undertaken with two aims in mind: (a) to ascertain whether theory would predict an absolute lower limit on the fiber diameter attainable in single-filament drawing operations, and (b) to determine the specific effect of various parameters upon this limiting value.

The beginnings of such a theoretical undertaking is presented below.

B. Governing Equations for Rod-Feed Drawing of Glass Fibers

The derivation presented below is developed specifically for the case of rod-feed drawing of glass monofilaments, although the general features of its results are probably applicable to the case of melt-drawing of glass fibers, as well. The reason for this choice was two-fold: first, this is the actual monofilament drawing operation developed and practiced during the current program and, second, this method lends itself much more clearly to a mathematical attack.

The conditions and choice of coordinate systems pertinent to rod-feed drawing of glass fibers are indicated in Fig. 1. The rod enters the hot zone of the furnace at a distance $s = 0$. As it passes through the furnace, it will be heated above its softening and flow points, and will begin to behave in a viscous fashion. The result of this will be a gradual drawing down of the rod diameter until, at the bottom of the furnace, the glass emerges in the form of a fine fiber.

For the geometry shown in Fig. 1, the following conditions will govern the behavior of the glass at an arbitrary section in the hot zone of the furnace. From volume constancy (preservation of mass)

$$r_o^2 v_o = r^2 v \tag{1}$$

where

v = fiber drawing velocity, ips

r = fiber radius, in.

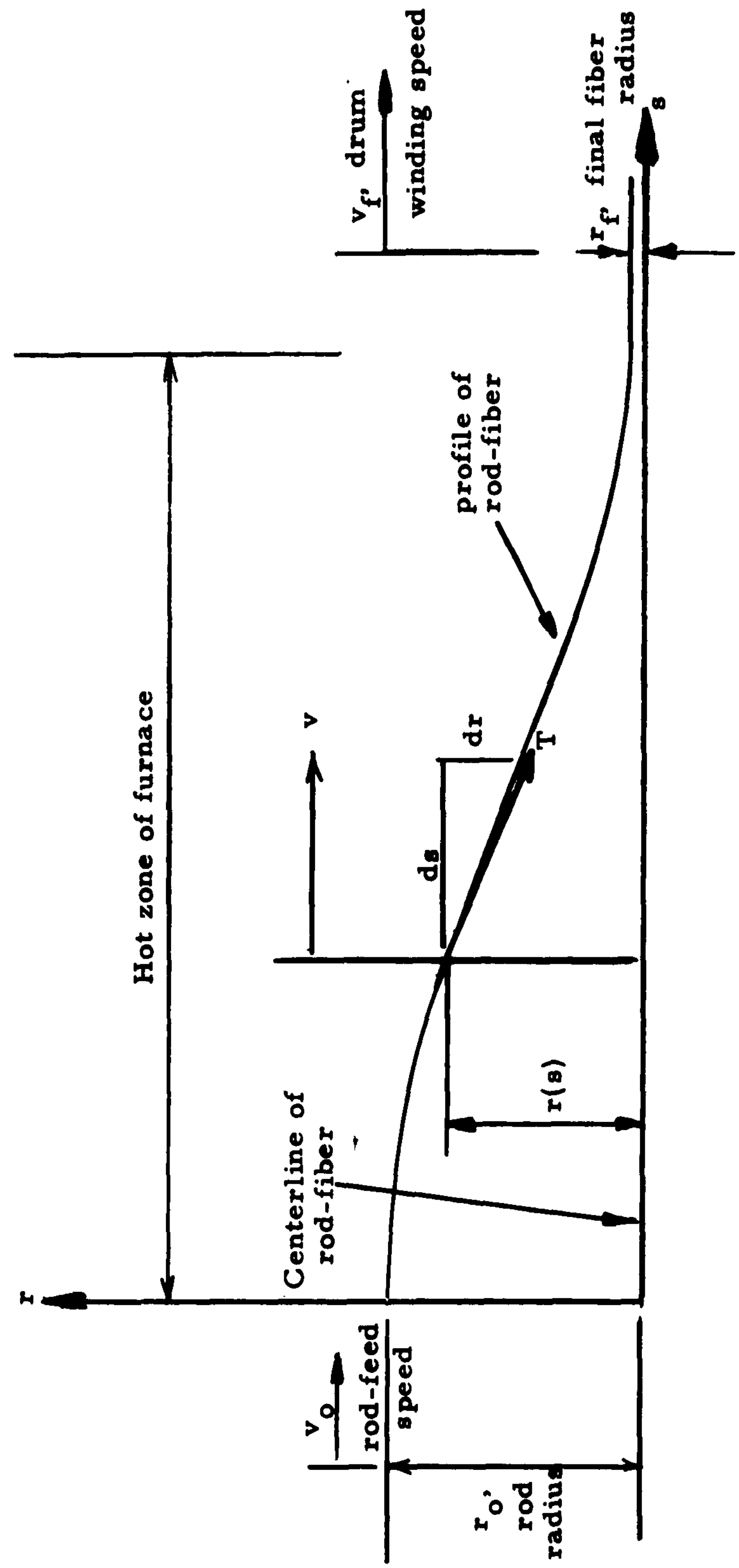

Fig. 1 SCHEMATICS OF THE GEOMETRY OF FIBERGLASS DRAWING FROM ROD FEED

and the subscript o denotes quantities in the original rod (before entry into the furnace). The relationship between strain and velocity can be established by reference to Fig. 2. If, over an infinitesimal distance ds, the back end of an elementary volume of the rod moves at a velocity v, and the front end of this volume element (which had undergone stretching by an amount of $d\epsilon$ per unit length) moves at a velocity of $v + dv$, then the following relationships hold for an infinitesimal time interval, dt:

Back end of elementary volume

$$v = ds/dt$$

Front end of elementary volume

$$v + dv = (1 + d\epsilon)\, ds/dt$$

and, subtracting the first from the second relationship, $dv = d\epsilon\; ds/dt$, or

$$d\epsilon/dt = \dot{\epsilon} = dv/ds \qquad (2)$$

where the overdot stands for differentiation with respect to time. Next, taking a force balance in the axial direction, as shown in Fig. 1,

$$\pi r^2 \sigma + \pi r S \left[1 + (dr/ds)^2 \right]^{-1/2} = F \qquad (3)$$

where

σ = axial stress in rod, psi
S = surface tension, lb/in.
F = drawing force, lb

The last remaining relationship concerns the rheological creep behavior of glass at elevated temperatures. While it is possible to postulate many reasonably valid creep laws, the one to be adopted here, because of

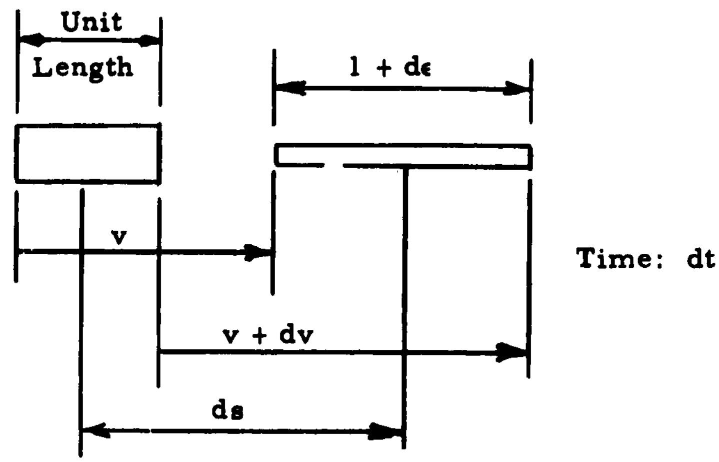

Fig. 2 SCHEMATICS OF RELATIONSHIP BETWEEN AXIAL STRAIN AND FLOW VELOCITY

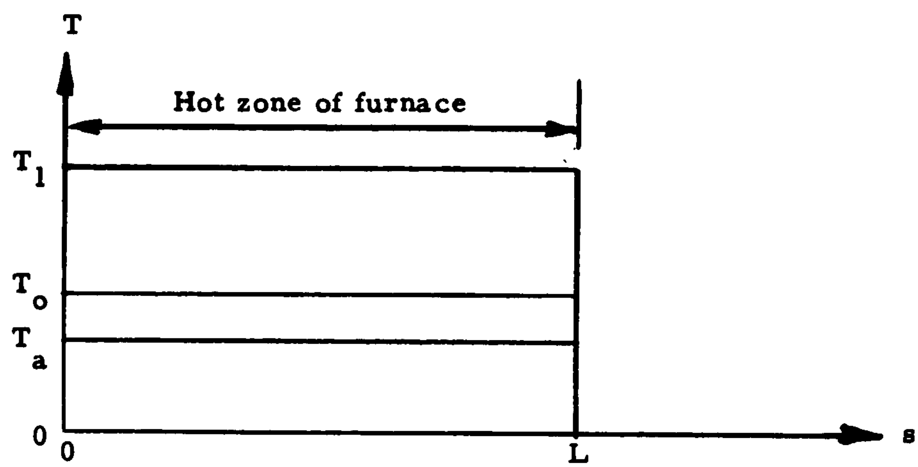

Fig. 3 ASSUMPTION FOR UNIFORM FURNACE TEMPERATURE DISTRIBUTION

its broad adaptibility and mathematical simplicity, is the relationship formulated by Nadai in the form*

$$\dot{\epsilon} = A\sigma^n \qquad (4)$$

where A is a temperature dependent quantity, and the value of n generally ranges between 3 and 5. It is worthwhile to note that, for n = 1, Eq. 4 reverts to the simple case of Newtonian viscosity

$$\dot{\epsilon} = \sigma/\eta \qquad (5)$$

where $\eta = 1/A$ is the conventional coefficient of viscosity commonly employed in hydrodynamics.

Equations 1 to 4 define a self consistent system of four equations with four independent variables (F, v, T and s; T being the temperature), with r (or ϵ) as dependent variables, it being assumed that the S = S(T) and A = A(T) functions are known. By successive substitution of Eqs. 4 and 2 into Eq. 3, it is then possible to write

$$r^2 (dv/Ads)^{1/n} + rS \left[1 + (dr/ds)^2 \right]^{-1/2} = F/\pi \qquad (6)$$

But, by Eq. 1

$$dv = -2r_o^2 v_o \, dr/r^3$$

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* Actually, Eq. 4 represents a simplification of multiaxial creep behavior to uniaxial conditions. The multiaxial case can be treated by adopting effective stress and strain relationships in the form

$$\tau = \frac{1}{\sqrt{2}} \left[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 \right]^{1/2}$$

$$\gamma = \frac{\sqrt{2}}{3} \left[(\epsilon_1 - \epsilon_2)^2 + (\epsilon_2 - \epsilon_3)^2 + (\epsilon_3 - \epsilon_1)^2 \right]^{1/2}$$

where the constant factors have been so chosen that for the uniaxial case ($\sigma_2 = \sigma_3 = 0$; $\epsilon_2 = \epsilon_3 = -\epsilon_1/2$) the effective stress and strain simplify to $\tau = \sigma_1$ and $\gamma = \epsilon_1$.

and substituting this result into Eq. 3 yields

$$r^2 (-2r_o^2 v_o \, dr/Ar^3 \, ds)^{1/n} + rS \left[1 + (dr/ds)^2 \right]^{-1/2} = F/\pi \quad (7)$$

Equation 7, representing the exact conditions of rod-feed fiber drawing, is a differential equation of substantial complexity. In order to obtain a physical representation of its implications, let us assume in the first approximation that the effect of surface tension is negligible as compared to the existence of the drawing force. Thus, by setting $S = 0$ into Eq. 7, there results the mathematically much more tractable expression of

$$-2r_o^2 v_o \, dr/Ar^3 \, ds = (F/\pi r^2)^n \quad (8)$$

Separating variables and integrating

$$-\frac{2\pi^n r_o^2 v_o}{F^n} \int_{r_o}^{r} r^{2n-3} \, dr = \int_o^s A \, d s \quad (9)$$

assuming that the drawing force is constant during the operation. This result can be integrated to

$$\frac{v_o}{n-1} \left(\frac{\pi r_o^2}{F} \right)^n \left[\frac{r}{r_o}^{2(n-1)} -1 \right] = - \int_o^s A \, d s, \quad (10)$$

or, after some rearrangement,

$$\left(\frac{r}{r_o} \right)^{2(n-1)} = 1 - K_1 \int_o^s A \, d s \quad (11)$$

where

$$K_1 = \frac{n-1}{v_o} \left(\frac{F}{\pi r_o^2} \right)^n .$$

Further derivations beyond this point must adopt some suitable mathematical assumption for the creep constant, A.

C. Assumption for Creep Constant

Strictly speaking, no explicit relationships are available for the temperature dependence of the creep constant, A, under assumption of the power creep law embodied in Eq. 4; the normal method of approach consisting of assuming that the $A = A(T)$ relationship is governed by an Arrhenius-law. However, under assumption of simple Newtonian viscosity, an empirical form of the viscosity-temperature relationship was formulated by Fulcher[1] in the form

$$\log \eta = -B_1 + \frac{B_2}{T - T_o} \tag{12}$$

where B_1, B_2 and T_o are constants, and T is the absolute temperature. Specific curves of viscosity vs. temperature for various glass compositions are given by Shand[2]. Naturally, for $T_o = 0$, Eq. 12 reverts to the simple Arrhenius law. Adopting this rather flexible expression to represent the functional dependence of the creep coefficient on temperature, one can state

$$\frac{1}{A} = B_1 \, e^{B_2/(T-T_o)} \tag{13}$$

Inserting this relationship into Eq. 11, there obtains

$$\left(\frac{r}{r_o}\right)^{2(n-1)} = 1 - \frac{K_1}{B_1} \int_o^s e^{-B_2/(T-T_o)} \, ds \tag{14}$$

Explicit results can now be obtained for various assumptions of the $T = T(s)$ relationship describing the temperature profile existing in the furnace.

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(1) Fulcher, G. S., J. Amer. Cer. Soc., 8, 339 (1925).

(2) Shand, E. B., "Glass Engineering Handbook", pp. 20-21, McGraw Hill Book Co., New York, N. Y. (1958).

D. Solutions of Fiber Shape for Various Furnace Temperature Profiles

Because of the comparative recency of this derivation, only two furnace temperature profiles were investigated to date, represented by the simple cases of the constant and linearly increasing temperature distributions.

1. Constant Furnace Temperature Profile

The temperature profile for this completely simplistic case is shown in Fig. 3, where T_a is the ambient temperature, T_o the value below which viscous flow cannot occur and T_1 is the constant furnace temperature, assumed to be representative of the fiber temperature as well. For this case Eq. 14 yields

$$\left(\frac{r}{r_o}\right)^{2(n-1)} = 1 - \frac{K_1}{B_1} s e^{-B_2/(T_1 - T_o)} \quad . \tag{15}$$

By token of Eq. 1 and substituting the above result

$$v = \left(\frac{r_o}{r}\right)^2 v_o = v_o \left[1 - \frac{K_1}{B_1} s e^{-B_2/(T_1 - T_o)}\right]^{-1/(n-1)} \tag{16}$$

and, from Eq. 2

$$\dot{\epsilon} = \frac{dv}{ds} = \frac{v_o}{n-1} \frac{K_1}{B_1} e^{-B_2/(T_1 - T_o)} \times \left[1 - \frac{K_1}{B_1} s e^{-B_2/(T_1 - T_o)}\right]^{-\frac{n}{n-1}} \tag{17}$$

Then, by Eq. 4

$$\sigma = \left[\frac{v_o}{n-1} \frac{K_1}{B_1 A} e^{-\frac{B_2}{T_1 - T_o}} \right]^{\frac{1}{n}} \times \left[1 - \frac{K_1}{B_1} s e^{-\frac{B_2}{(T_1 - T_o)}} \right]^{-\frac{1}{n-1}} \tag{18}$$

or, after substitution for A from Eq. 13

$$\sigma = \left(\frac{v_o K_1}{n-1}\right)^{1/n} \left[1 - \frac{K_1}{B_1} s e^{-B_2/(T_1 - T_o)} \right]^{-1/n-1)} . \tag{19}$$

Combining now Eqs. 16 and 19 yields

$$\sigma = \left(\frac{K_1}{n-1}\right)^{\frac{1}{n}} v_o^{(\frac{1}{n} - 1)} v = \frac{F}{\pi r_o^2} \frac{v}{v_o} = \sigma_o v / v_o . \tag{20}$$

The following additional results can be written down by use of Eqs. 1, 3 and 16

$$r = r_o (v_o / v)^{1/2} \tag{21}$$

$$F = \pi r_o^2 \left(\frac{K_1 v_o}{n-1}\right)^{1/n} \tag{22}$$

$$v = v_o (1 - K_1 A s)^{-\frac{1}{n-1}} \tag{23}$$

where v is the filament velocity at any arbitrary point at a distance s down from the top of the furnace, while the final drawing velocity of the fiber emerging from the furnace (the take-up speed of the winding drum) is given by

$$v_f = v_o (1 - K_1 A L)^{-\frac{1}{n-1}} \tag{24}$$

where L is the length of the heated zone.

Although the significance and meaning of Eqs. 20 to 24 will be discussed later, is interesting to note here that these equations are independent of the axial location of the rod-fiber continuum, and hold for any particular section within and without the furnace.

2. Linearly Increasing Furnace Temperature Profile

The conditions covering this case are shown in Fig. 4; once again, it will be assumed that the furnace and fiber temperatures are identical. The effective temperature distribution will now be given by

$$T = T_a + (T_1 - T_a)\frac{s}{L} \qquad 0 \leq s \leq L \tag{25}$$

so that Eq. 14 will take the form

$$\left(\frac{r}{r_o}\right)^{2(n-1)} = 1 - \frac{K_1}{B_1}\int_{s_1}^{s} e^{-\left(\frac{B_2}{C_1+C_2 s}\right)} ds \tag{26}$$

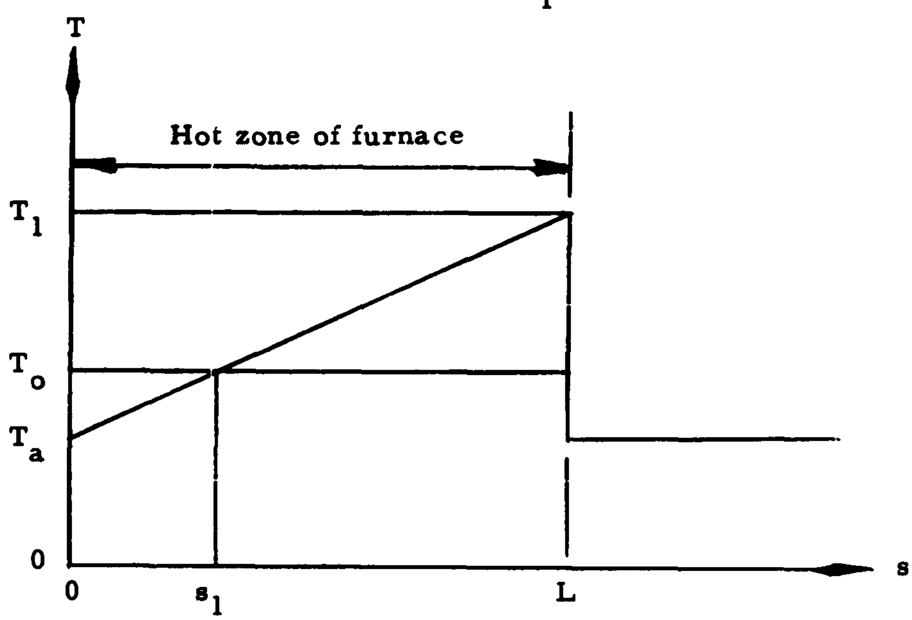

Fig. 4 ASSUMPTION FOR LINEARLY INCREASING FURNACE TEMPERATURE PROFILE

where

$$s_1 = \frac{T_o - T_a}{T_1 - T_a} L$$

$$C_1 = T_a - T_o \qquad (27)$$

$$C_2 = \frac{T_1 - T_a}{L}$$

Letting now

$$\zeta = - \frac{B_2}{C_1+C_2 s} = - \frac{B_2}{T_a - T_o + (T_1 - T_a)\frac{s}{L}}$$

so that

$$s = -\frac{1}{C_2} \left(\frac{B_2}{\zeta} + C_1 \right) \quad \text{and} \quad ds = \frac{B_2 d\zeta}{C_2 \zeta^2}$$

and using this transformation in Eq. 26, there obtains

$$\left(\frac{r}{r_o} \right)^{2(n-1)} = 1 - \left(\frac{K_1 B_2}{C_2 B_1} \right) \int_{\zeta_1}^{\zeta} e^{\zeta} \zeta^{-2} d\zeta \qquad (28)$$

where

$$\zeta_1 = - \frac{B_2}{C_1 + \frac{T_o - T_a}{T_1 - T_a} C_2 L} = -\infty \qquad (29)$$

Equation 28 can be integrated into the form of a logarithmic integral function or, alternately, into the infinite series expression

$$\left(\frac{r}{r_o}\right)^{2(n-1)} = 1 - \left(\frac{K_1 B_2}{C_2 B_1}\right)$$

$$\times\left[-\frac{e^{\zeta}}{\zeta} + \log\zeta + \zeta + \frac{\zeta^2}{2\cdot 2!} + \frac{\zeta^3}{3\cdot 3!} + \ldots +\right]_{\zeta_1}^{\zeta} \qquad (30)$$

If the final (nascent) fiber diameter is of interest, such can be obtained by using the upper limit of

$$\zeta_L = -\frac{B_2}{C_1+C_2L} \qquad (31)$$

in Eq. 30. The derivation has not been yet carried beyond this step to the point where explicit relationships for fiber diameter, stress and drawing force as functions of the drawing velocity could be derived for the linear temperature distribution.

E. Discussion and Conclusions

Although to date only the extremely simplistic temperature distribution of a uniform axial furnace temperature profile has been fully evaluated, this case has already yielded an extremely valuable insight into the mechanics of fiber drawing from rod feed.

Examining the results of the simplest case of a uniform furnace temperature profile, as given by Eqs. 20 to 24, one arrives at the following conclusions:

a. The stress in the nascent fiber is directly proportional to the drawing speed, as shown by Eq. 20.

b. The radius of the nascent fiber is inversely proportional to the square-root of the drawing speed, as exhibited by Eq. 21.

c. A, the inverse of viscosity, is a very small number, but increases rapidly as the temperature rises. Therefore, the factor multiplying v_o in Eq. 24 (the bracketed term raised to the -1/(n-1) power)

rises rapidly from the value of 1 as the temperature rises above T_o. Thus, as would be expected, the ratio of nascent fiber velocity to rod-feed speed increases rapidly as the furnace temperature is increased.

Before passing, it is of substantial interest to note that if the simplification of Newtonian viscosity is invoked with its corresponding characterization of $n = 1$, the solution proceeds to go "ad absurdum"; v_f rises to infinity (note Eq. 24, observing that the value of the bracket is less than one), and the drawing stress (Eq. 20), and hence the drawing force, vanishes. The case thus correctly represents a liquid, which will just drop out of the furnace at no drawing stress and at an infinite speed as $n \rightarrow 1$.

The real significant results of this derivation are those contained in points (a) and (b). That is, to repeat, as the drawing speed increases, the fiber drawn becomes finer, but the stress in it increases. Obviously, this cannot continue ad infinitum. Even though the strength of the fiber increases as its diameter is reduced, this increase is generally hyperbolic and, in the size range of question, far slower than linear. Moreover, the diameter is reduced only with the square root of the drawing speed, while the stress in it rises linearly with winding velocity.

Without much question, at some drawing speed the point must come at which the drawing stress outruns the more slowly increasing fiber strength. At this point rupture of the monofilament will occur; the diameter corresponding to this limit will then represent the finest fiber that can be drawn at the furnace setting in question.

These conclusions are preliminary and, at this time, unsupported by numerical calculations. It should be recalled that they are based on several simplifying assumptions: (a) the effect of surface tension has been ignored and (b) the furnace temperature is assumed to be identical with the fiber temperature.

Furthermore, the only completed derivation refers to the simplistic assumption of a uniform furnace temperature. Nevertheless, results already obtained clearly indicate the reasons for the frequently observed

condition that there is a very real limitation of fiber size, such that no fibers below a certain diameter can be drawn in monofilament winding operations.

III. FIBER DRAWING EXPERIMENTS

The fiber drawing experiments conducted during this research period were a continuation of the efforts to improve the metal coating procedure for glass fibers and to continue the efforts of determining the feasibility of the bundle drawing technique in producing ultra-fine glass filaments. The glass employed in the metal coating experiments was an aluminosilicate type, Corning Glass Works Code No. 1723 glass. The metallizing alloys were the same as those employed during the previous research period(3). They were an aluminum-silicon alloy (95 Al - 5 Si) and a lead-indium alloy (80 Pb - 20 In).

A. Metallizing Experiments

The metallizing experiments were conducted during this research period in order to provide a quantity of metal coated glass fibers to be used in a subsequent bundle redrawing operation. Therefore, the aim was to provide relatively heavy (25 to 50 micron) fibers with a fairly uniform metallic coating such that upon redrawing the fibers would be prevented from fusing together by the presence of the metallic coating. The metallizing experiments performed during this research period are summarized in Table I. The first two coating attempts shown in Table I were made with the aluminum silicon alloy. Previous experience in the redrawing of glass fibers coated with the aluminum silicon alloy, as reported earlier(4), indicated that perhaps an inert atmosphere was required in the redrawing of the aluminum silicon coated fibers in order to prevent the severe oxidation of the coating which took place when the redrawing was performed in air. Therefore, an additional supply of aluminum silicon coated glass fibers

(3) Weil, N. A., Islinger, J. S., et al, "Investigation of Glass Fiber Strength Enhancement Through Bundle Drawing", Third Quarterly Report to BuWeps, Nov. 1961, p. 10.

(4) ibid, p. 14.

Table I

RESULTS OF METAL COATING EXPERIMENTS ON ALUMINOSILICATE (1723) GLASS

Furnace Temperature[1]								
upper air, °F	middle t.c., °F	Rod Feed in./min	Drum Speed rpm	Metallizing Alloy	Crucible Temp °F	Atmosphere	Fiber diameter microns	Remarks
1850	2115	0.23	200	95 Al - 5 Si	1290	Air	20 - 25	Excessive coating[3], fibers curled.
1880	2115	0.23	200	95 Al - 5 Si	1240	Air	25	Continuous coating but non-uniform thickness.
1880	1985	0.23	100	80 Pb - 20 In	620	Air	50 - 60[2]	Continuous coating, heavy on one side.
1860	1985	0.23	400	80 Pb - 20 In	575 - 600	Argon	25 - 35	No coating.
1800	1985	0.23	300	80 Pb - 20 In	540	Argon	25 - 35	No coating.
1800	1985	0.23	300	80 Pb - 20 In	560	Argon	25 - 35	No coating.
1860	1985	0.23 - 0.26	200 - 400	80 Pb - 20 In	560 - 625	Air	50 - 60[2]	For bundle re-draw.

Notes: 1 Lower furnace element was maintained at 20% of maximum input. This amounts to air temperatures ranging from 800 to 1700°F depending upon inputs to upper and middle elements. For the experiments summarized in this table the air temperature in the lower zone was about 1200°F.

2 Coated diameter.

3 Coating thickness approximately 5 to 8 microns.

was prepared for the subsequent redraw operation using an inert atmosphere. Even though the coating technique has not been perfected for the aluminum silicon alloy, as evidenced by the fact that the coating appears to be applied to only one side of the fiber, the fibers so coated were employed in a bundle redraw operation described later in this report.

The remaining metallizing experiments were cond :cted with the lead indium alloy. The first such metallizing experiment reported resulted in a coated fiber with a continuous coating heavier on one side than on the other with a resultant fiber diameter of 50 to 60 microns. This coating experiment was conducted in air. The enxt coating experiment was conducted using an argon atmosphere as follows: when a fiber of the proper diameter was started, the cover plate was put into place closing the metallizing unit. Bottled lamp grade argon was permitted to flow into the metallizing unit and exited upward through the furnace. The molten metal contained in the crucible was then brought close to the fiber until contact was made between the fiber and the molten metal bead. It appeared that coating of the fiber then took place. However, upon examination of the fiber upon conclusion of the experiment, it was noted that the fibers had not been coated with the lead indium alloy. Several other attempts were made to apply a coating of this alloy in the inert argon atmosphere with slight variations in the temperature of the molten metal but these were to no avail either. Finally, another set of lead indium coated glass fibers were prepared in an air atmosphere. These were similar to the others in that a fairly uniform coating was deposited and resulted in a fiber with a maximum overall diameter of 50 to 60 microns.

B. Bundle Drawing Experiments

The next series of experiments conducted during this research period were the bundle drawing experiments. The object of these bundle drawing experiments was to determine under what conditions it is possible to redraw and retain discrete glass filaments with ultra fine diameters. In the bundle drawing process, hundreds of coated or uncoated glass fibers are bundled and collected in a glass tube or sheathing. This glass tube-filament combination is drawn into a fiber in the same manner that the glass

rods are drawn into a fiber. The diameter of the resulting redrawn fiber is a function of the rate at which the glass tube-filament combination is fed into the furnace, the furnace temperature, and the drawing speed. A redrawn fiber with the diameter of the order of 25 to 50 microns is desired as was mentioned earlier. If the initial rod is 1/4 in. in diameter and if the filament drawn therefrom is 25 microns in diameter before coating, this would result in a first drawing diameter reduction of about 250 times. Thus, if the bundled fibers are collected in a sheathing of 1/4 in. diameter and the same reduction is applied, there would result a fiber with a diameter of 25 microns containing filaments within, with a diameter of 0.1 micron.

Results of the bundle drawing experiments are summarized in Table 2. The first such bundle drawing experiment during this period was conducted with 15 micron diameter uncoated E-glass fibers bundled into an aluminosilicate (Corning Glass Works Code No. 1720) glass tube with an outside diameter of 0.25 in. and with a wall thickness of 0.026 in. The E-glass fibers employed were drawn previously from a 1/4 in. diameter E-glass rod. The fibers were collected and drawn into the glass tube by hand as tightly as possible. The resulting fiber from the bundle drawing operation had a minimum diameter of 100 microns. When a cross section of the fiber was viewed under the microscope, it was noted that the E-glass fibers had fused into a solid mass within the tube of reduced diameter and there was also an air space. A drawing of the cross section is given in Fig. 5.

The next bundle drawing experiment was performed with 100 micron diameter 1723 (aluminosilicate) glass fibers bundled in a 1/4 in. diameter thin wall 1720 glass tubing. Under the original temperature settings as noted in Table 2, a long period of time elapsed with no starting bulb emerging from the furnace. The temperature settings were increased and when the starting bulb appeared, it was noted that the starting bulb contained many air bubbles and that the individual fibers appeared to be fused together. Attempts were made to start the fiber on the take-up drum but these were to no avail because the fiber broke each time it was started on the drum. Another attempt was made to redraw with the same fibers and glass tubing

Table 2

RESULTS OF BUNDLE RE-DRAWING EXPERIMENTS (SEVERAL GLASSES, COATINGS)

Material (fibers)	Furnace Temperature upper Air, °F	Furnace Temperature middle Air, °F	Rod Feed in./min	Drum Speed rpm	Atmos-phere	Fiber diameter microns	Sheath Condition	Remarks
E-glass	1500	1860	0.23	300	Air	100	Open tube	Filaments fused.
1723 glass	1600	1725+	-	-	Air	No fiber	Open tube	Could not start fiber on take-up drum.
1723 glass	1700 - 1850	1725	-	-	Air	No fiber	Open tube	Could not start fiber on take-up drum.
E-glass	1600	1740	-	-	Air	No fiber	Open tube	Could not start fiber on take-up drum.
E-glass and 1723 glass	1750 - 1840	1740	0.30	100	Air	110	Open tube	Only short length obtained.
1723 coated w. 95 Al - 5 Si	1800	1740+	-	-	Argon	No fiber	Open tube	Coating oxidized.
1723 coated w. 80 Pb - 20 In	1600	1860	0.35	140 - 400	Air	70 by 30	Sealed tube	Oblong ribbon-like fiber.
1723 coated w. 80 Pb - 20 In	1600	1860	0.35	140 - 400	Air	60	Open tube	Cylindrical fiber.
1723 coated w. 80 Pb - 20 In	1600	1750 - 1785	0.31	40 - 50	Air	150 - 200	Open tube	
1723 coated w. 80 Pb - 20 In	1600 1600	1860 1910	0.32 0.32	200 - 400 400	Air Air	35 - 45 20 - 33	Open tube Open tube	Fiber broke Continuous fiber
1723 coated w. 80 Pb - 20 In	1600	1965	0.32	400	Air	20 - 44	Sealed tube	Continuous until fiber broke
1723 coated w. 80 Pb - 20 In	1600 1600 1600 1600	1910 1890 1890 1890	0.35 0.35 0.35 0.35	400 225 200 400	Air Air Air Air	60 60 60 60	Sealed tube Sealed tube Sealed tube Sealed tube	Fiber broke Fiber broke Fiber abrading and broke Fiber broke

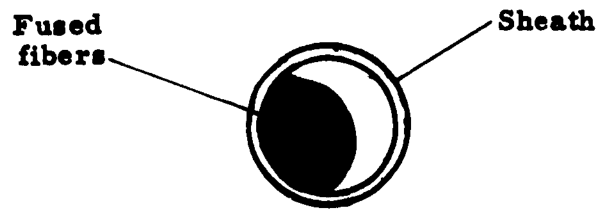

Fig. 5 CROSS SECTION OF RE-DRAWN UNCOATED E-GLASS FIBERS BUNDLED IN 1720 GLASS TUBE. (Fibers fused together)

Fig. 6 SECTIONS OF RE-DRAWN BUNDLED FIBERS, SHOWING FUSION OF FIBERS AND NODULARIZING OF COATING METAL, X500. (Aluminosilicate, 1723 glass fibers coated with Pb-In alloy purged with argon, evacuated and sealed in 1720 sheath)

Fig. 7 SECTIONS OF RE-DRAWN BUNDLE FIBERS, X500. (Conditions as in Fig. 6 except bundle was neither argon purged nor evacuated)

as were employed in the previous attempt. The results of this next bundle drawing experiment were the same as the previous attempt.

In the fourth bundle drawing experiment a 1/4 in. thin wall 1720 glass tubing was filled with as many E-glass fibers as possible. The diameters of the E-glass fibers ranged from 25 to 35 microns. In order to get the maximum amount of fibers inside the tube it was necessary to twist the fibers, which resulted in numerous fiber failures during the bundling process. At the temperatures noted the starting bulb came down very slowly and it was noted that the force required to draw the fiber was greater than the fiber could tolerate; before a fiber could be started on the drum it broke. It was observed that individual fibers within the bulb had fused together and therefore it was surmised that this was true within the redrawn fiber as well.

In the fifth bundle drawing experiment a combination of uncoated 25 to 35 micron diameter E-glass and 1723 glass fibers were assembled and drawn into a 1/4 in. thin wall 1720 glass tubing. A starting bulb lowered and broke several times before a fiber could be started on the take-up drum. A quantity of fibers with a minimum diameter of 110 microns were produced. However, because of the apparent unsymmetrical contraction of the individual filaments within the fiber (composed of a combination of E-glass and 1723 fibers) the redrawn fiber when removed from the drum had some curl. It was observed that E-glass filaments had fused to one another and the 1723 glass filaments had fused to each other also. It became evident from this and previous bundle drawing experiments that even at the relatively low drawing temperatures in which only softening (rather than melting) of the glass takes place, fusion of individual fibers occurs. Therefore, in order to assure that individual filaments will retain their identity after the bundle or redrawing operation, it is necessary that the fibers be coated either with the metallic coating or with glass of another composition so that the fusion of individual filaments cannot take place.

The sixth bundle drawing experiment, therefore, employed 1723 glass fibers coated with the 95 Al - 5 Si alloy collected in a 1/4 in. thin

wall 1720 glass tube. In a previous bundle drawing experiment[5] in which aluminum-silicon coated fibers were employed severe oxidation took place and a fiber could not be started. Therefore, in this attempt the drawing was conducted in an argon atmosphere. In spite of this, difficulties were experienced in starting a fiber and the same type of oxidation was noted, namely the fibers were blackened and appeared to be very brittle. Another attempt was made with the same combination of coated fibers and tubing and the result was the same.

As an explanation for the above behavior, it is believed that the aluminum-silicon coating reacted with oxygen adsorbed from the glass. It was further theorized that this type of reaction could not take place with the other alloy coating which has been employed on this program (the lead-indium alloy). Therefore, a series of bundle drawing experiments employing this latter coating were undertaken. In preparation for this series of experiments a quantity of 1723 glass fibers were coated with the lead-indium (80 Pb - 20 In) alloy with an overall coated diameter of 50 to 60 microns. These fibers were collected and bundled in six 1/4 in. 1720 thin wall glass tubes. Three of these stuffed tubes were alternately purged with argon gas and evacuated several times and both ends of each tube were sealed upon the final evacuation. The other three tubes received no such treatment.

The first tube and fiber combination subjected to the bundle drawing experiment was selected from the group which had been purged with argon, evacuated and sealed. Before the starting bulb emerged from the furnace, it was noted that the portion of the tube within the heated zone of the furnace had collapsed when its temperature reached the softening zone. No difficulties were encountered in starting and drawing a fiber; it appeared as though the bundle drawing operation had at last been successful. However, upon inspection of cross sections of the resulting fibers, it was noted that the fibers had an oblong shape with a maximum dimension of about 70 microns in the long axis and 25 to 50 microns in the short axis. More significant was the fact that the metallic coating had dewetted from the glass and permitted individual filaments to fuse together while the metal itself collected

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(5) loc. cit.

in globules throughout the length of the fiber. No oxidation of the metallic coating took place. A photomicrograph containing several sections of one of the fibers taken at a magnification of 500 times is given in Fig. 6.

Since the first of this series of experiments resulted in no oxidation of metallic coating material in a protective vacuum environment, it was decided that the influence of an air atmosphere would be explored. Thus, the next experiment was with one of the tube fiber combinations which had not been evacuated. Again, no difficulties were encountered in starting the fiber and this time a fiber with a circular cross section with a diameter of 60 microns was obtained. A photomicrograph of one of these samples taken at a magnification of 500 times is given in Fig. 7. No oxidation of the metallic coating was observed and again the metallic coating dewetted the glass fibers and permitted individual filaments to fuse together while the metal itself collected in globules throughout the length of the fiber.

The remaining four tube-fiber combinations of this series were also subjected to the bundle drawing experiments with variations in the furnace temperature and in the drawing speed to produce some equivalent and some finer fibers. The minimum fiber diameter attained in this series of experiments was 20 microns. The metallic coating always behaved in the same manner namely dewetting and permitting the individual filaments to fuse together and collecting in globules throughout the length of the fibers. Oxidation of the lead-indium coating material did not take place.

Most of the fiber drawing experiments conducted during this research period were performed with the 1723 aluminosilicate glass instead of the E-glass because of the unavailability of the latter. However, during the time in which this report was prepared, a shipment of 50 E-glass rods was received from the Corning Glass Works. The rods had diameters slightly greater than 1/4 in. and were irregular in cross section and in diameter and had several surface flaws. Some of these rods will be employed in the "as received" condition, and some will be centerless ground to obtain a close tolerance 1/4 in. diameter rod. These rods will be employed in later experiments during the next research period.

IV. PLANS FOR FUTURE WORK

A. Analytical

The actual temperature distribution at the axis of the furnace has been obtained from several probe-traverse measurements; samples of these results are shown in Fig. 8.

Future work, therefore, will consist of

a. Obtaining numerical predictions for the simple case of the uniform temperature distribution.

b. Completing the derivation for the linear furnace temperature rise.

c. Developing solutions, if possible, for analytical furnace temperature distribution closely approximating the actual profile shown in Fig. 5.

These plans for future work should be tempered by the consideration that comparatively little time and resources remain to the completion of work on the current funding phase of this program.

B. Experimental

The experience in the bundle drawing experiments with dewetting of the metallic coating indicates that a reexamination of suitable coating materials must be conducted. Thus, during the remaining portion of this contract, examination of the dewetting phenomenon and of the possibility of using higher melting and, perhaps, vapor deposited metals as coatings will be undertaken. The lead-indium alloys will be studied with regard to dewetting temperature and the influence thereon of small additions of such metals as titanium, zirconium, silicon, and germanium. If time permits, the influence of ambient environment (air, inert gas, or vacuum) will be included. The vapor deposited metallic films will be considered both as complete deposits and as substrates for the presently used alloys applied as overcoats by the present technique.

The fiber drawing experiments will continue with efforts being directed toward the redrawing of glass fibers. In the absence of a suitable metallic coating material for preventing fusion of individual filaments within the redrawn fiber, attempts will be made to redraw glass fibers coated with glass

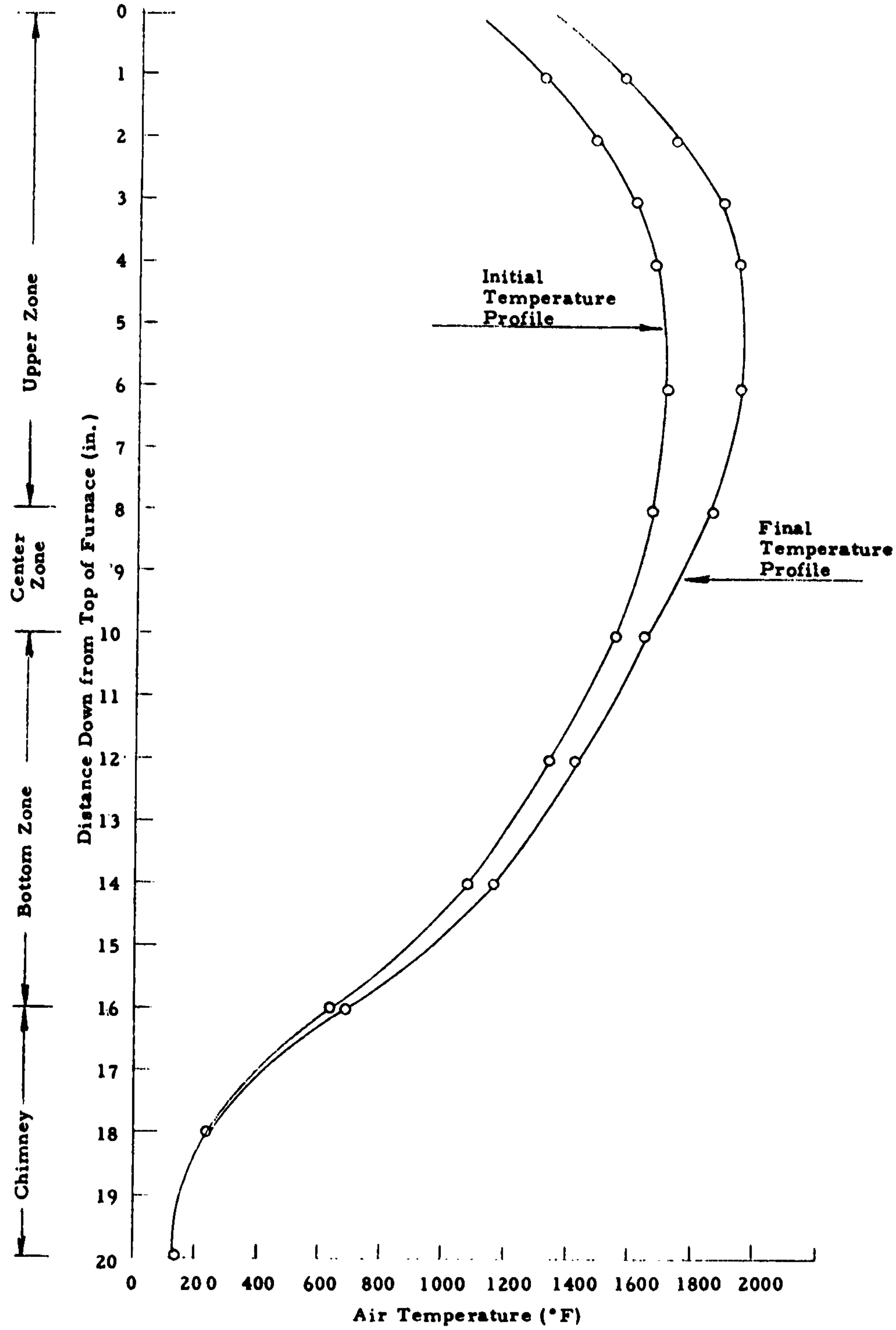

Fig. 8 **TEMPERATURE PROFILE IN FIBER DRAWING FURNACE UNDER CONDITIONS FOR PRODUCING 10μ E-GLASS FIBER**

of another composition. Other efforts will be expended in the direction of drawing, coating and redrawing E-glass fibers to assess the characteristics of this material in comparison with those of the 1723 glass. During the latter portion of the remaining research period it is hoped that strength determinations of some of the resulting fibers can be made in an attempt to verify some of the theoretical predictions of the analytical phase of this program.

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